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4-(*N*,*N*-Dimethylaminomethylene)-2-phenyl-2-oxazolin-5-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.055
wR factor = 0.163
Data-to-parameter ratio = 13.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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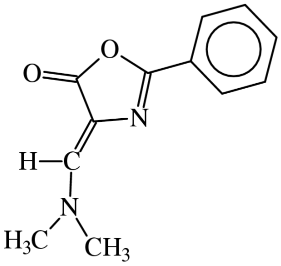
The title compound, $C_{12}H_{12}N_2O_2$, crystallizes in the triclinic space group $P\overline{1}$ with two crystallographically independent molecules in the asymmetric unit. These two molecules differ slightly in the relative orientation of the phenyl and oxazoline rings. The molecular packing in the crystal is stabilized by C—H···O hydrogen bonds and van der Waals interactions.

Comment

The X-ray investigation of the title compound, (I), was undertaken as a part of our study on the structure of the oxazolin-5-one derivatives (Vasuki *et al.*, 2001), and to study the effect of substituents at the 2- and 4-positions of the oxazoline ring.



(I)

The asymmetric unit consists of an enantiomeric pair of molecules with their centroid at (0.267, 0.232, 0.249) (Fig. 1). The corresponding bond distances and angles in this enantiomeric pair agree with each other, but the two molecules differ slightly in the relative orientations of the phenyl and oxazoline rings, with dihedral angles of 7.35 (1) and 1.92 (1)°. The dihedral angles between the oxazoline ring and the dimethyaminomethylene moiety are 2.20 (2) and 2.97 (2)° for the two molecules. The planarity of the oxazoline ring is not affected by the substituents at the 2- and 4-positions. The C6—N7 bond shows partial double-bond character (Table 1), owing to the delocalization of the lone pair of electrons of the N atom over the N7—C6═C4 moiety. The N3—C2—C10 exocyclic angle of 128.4 (2)° [128.1 (2)°, for the second molecule] is significantly greater than the normal value of 120°; this might be a consequence of repulsion between the lone pair of electrons on N3 and H11 attached to C11 (N3···H11 = 2.66 Å [2.63 Å]). In the crystal, the molecules exist as centrosymmetric C—H···O intermolecular hydrogen-bonded dimers (Table 2). A short intramolecular C—H···O contact is observed between C15 and O1, with an H15···O1 distance of 2.49 Å (2.50 Å).

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Experimental

The title compound was prepared by adding phosphorus oxychloride (2.7 g, 18 mmol) to a stirred solution of methyl hippurate (6 mmol) in dry DMF (8 ml). The resulting reaction mixture was heated at 363 K for 30 min, cooled and poured into crushed ice. After stirring for 30 minutes, a saturated aqueous solution of $NaHCO_3$ was added until the pH reached 7.0. The resulting precipitate was filtered and the filtrate was extracted with methylene chloride (3 × 20 ml). The organic phase was successively washed with water (3 × 30 ml) and saturated brine solution (30 ml). The extract was dried over anhydrous sodium sulfate (Na_2SO_4), filtered, evaporated *in vacuo* (rotatory evaporator) and the crude product was recrystallized from methanol.

Crystal data

$C_{12}H_{12}N_2O_2$
$M_r = 216.24$
Triclinic, $P\overline{1}$
$a = 8.416$ (6) Å
$b = 11.288$ (3) Å
$c = 12.097$ (3) Å
$\alpha = 85.39$ (3)°
$\beta = 73.97$ (4)°
$\gamma = 83.31$ (4)°
$V = 1095.6$ (9) Å^3
$Z = 4$
$D_x = 1.311$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10$–15°
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Plate, orange
0.60 × 0.15 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.947$, $T_{max} = 0.991$
4344 measured reflections
3849 independent reflections
2681 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 25.0°$
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 14$
2 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.163$
$S = 1.13$
3849 reflections
293 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0814P)^2 + 0.2456P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.008 (2)

Table 1
Selected interatomic distances (Å).

C2*A*—N3*A*	1.285 (3)	C2*B*—N3*B*	1.279 (3)
N3*A*—C4*A*	1.401 (3)	N3*B*—C4*B*	1.411 (3)
C4*A*—C6*A*	1.378 (3)	C4*B*—C6*B*	1.365 (4)
C4*A*—C5*A*	1.425 (3)	C4*B*—C5*B*	1.422 (4)
C5*A*—O5*A*	1.220 (3)	C5*B*—O5*B*	1.226 (3)
C6*A*—N7*A*	1.323 (3)	C6*B*—N7*B*	1.315 (3)
N7*A*—C9*A*	1.448 (3)	N7*B*—C8*B*	1.452 (3)
N7*A*—C8*A*	1.458 (3)	N7*B*—C9*B*	1.460 (3)

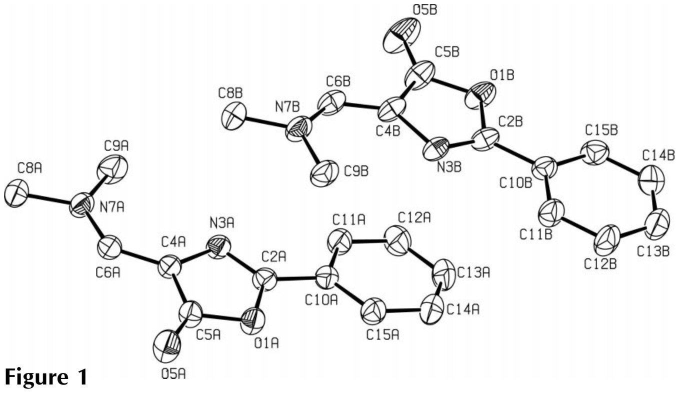

Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids. All H atoms have been omitted for clarity.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6*A*—H6*A*···O5*A*^{i}	0.93	2.39	3.287 (4)	161
C8*A*—H8*A*···O5*A*^{i}	0.96	2.45	3.365 (4)	160
C8*B*—H8D···O5*B*^{ii}	0.96	2.38	3.296 (5)	161

Symmetry codes: (i) $-1 - x, 1 - y, 1 - z$; (ii) $1 - x, 1 - y, -z$.

All H atoms were fixed geometrically and refined using a riding model, with C—H = 0.93–0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H and 1.2 $U_{eq}(C)$ for all others. One of the methyl groups (C9*B*) was found to be disordered; it was treated as an idealized disordered methyl group, with two positions rotated from each other by 60°, and the occupation factors fixed at 0.5.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP*97 (Zsolnai, 1997); software used to prepare material for publication: *SHELXL*97.

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